Mechanical Properties of Air Plasma Sprayed Environmental Barrier Coating (EBC) Materials

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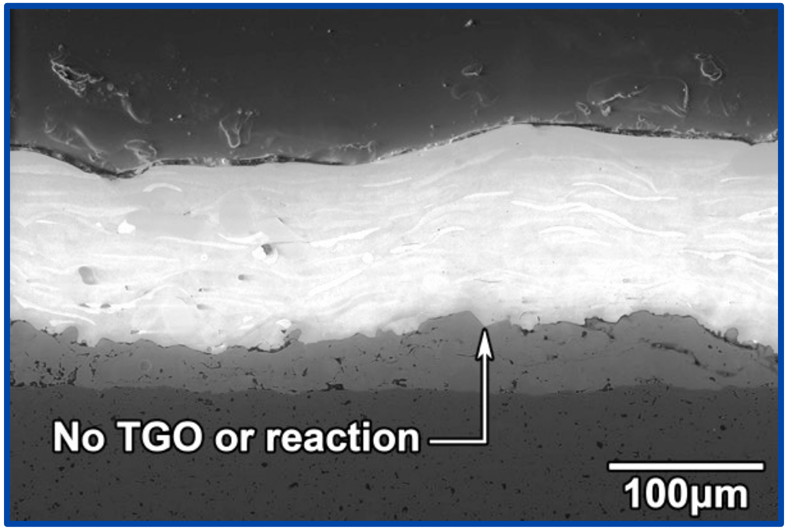
Outline: testing of relevant mechanical properties of APS EBCs: $Yb_2Si_2O_7$ and Si

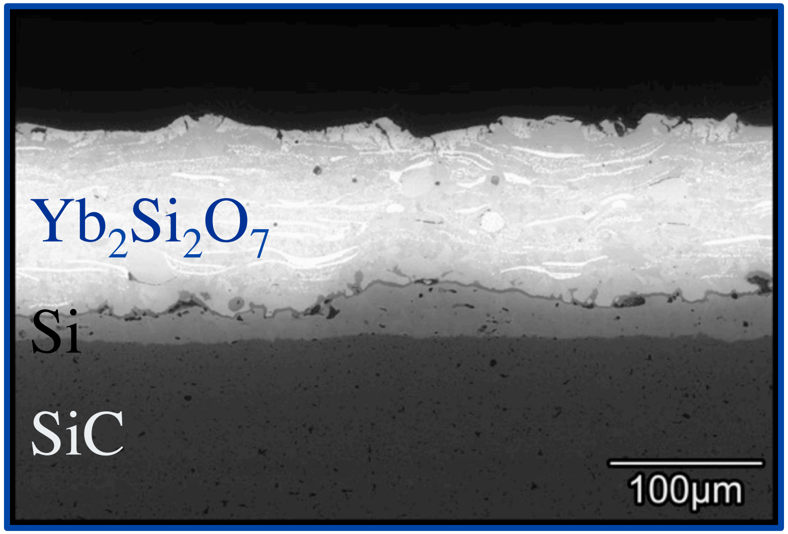
1. Very brief introduction
2. Methodology
3. APS $Yb_2Si_2O_7$ properties
    1. APS structure
    2. Elastic modulus, flexure strength, K_{IC}
    3. Isothermal creep
    4. Laser thermal gradient creep
4. APS Si properties
    1. APS structure
    2. Elastic modulus, flexure strength, K_{IC}
    3. Laser thermal gradient creep
    4. Interlaminar/interfacial toughness

$Yb_2Si_2O_7$/Si EBC Property Testing Goals

Annealed Coating



2000 Steam Cycles

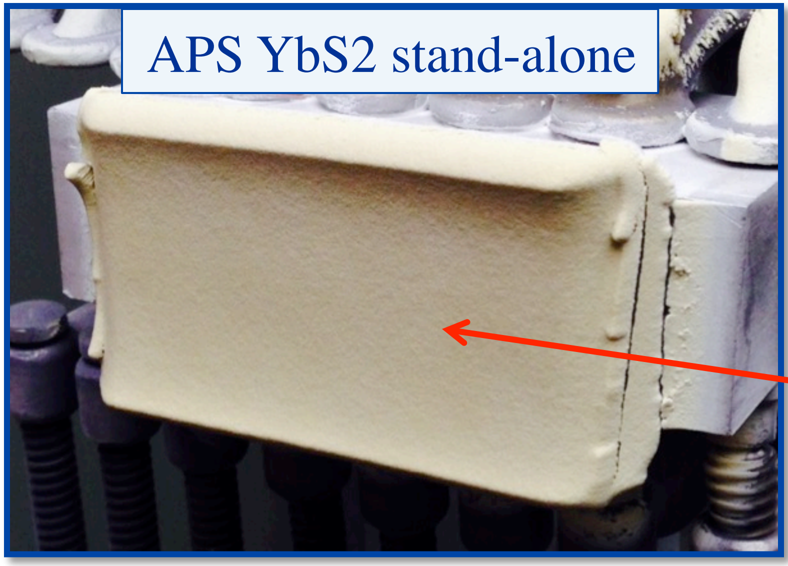


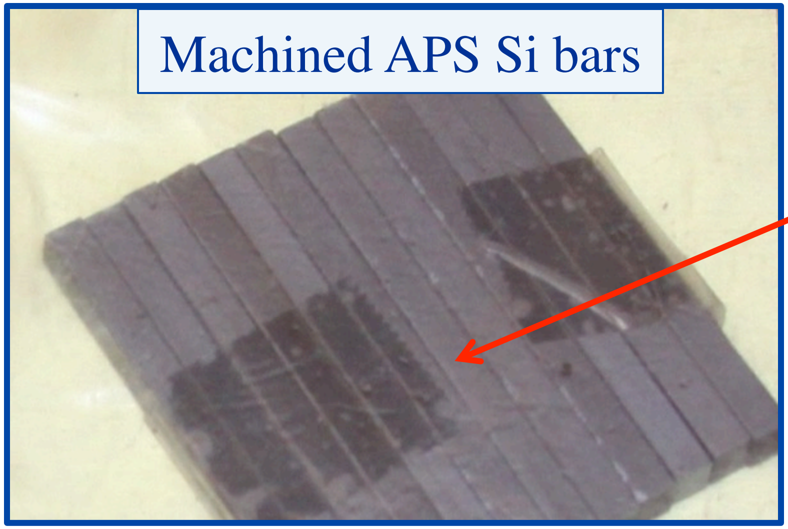
Provide insight into how APS processing can alter mechanical properties

Provide thermophysical data for modeling efforts of EBC systems

Understand mechanical properties of a relevant EBC system

Methodology





- Refine spray parameters
- Deposit APS stand-alone material:

  $Yb_2Si_2O_7$ and Si

  @ 130 x 65 x 13mm
- Machine (grind) APS stand-alone material into test bars

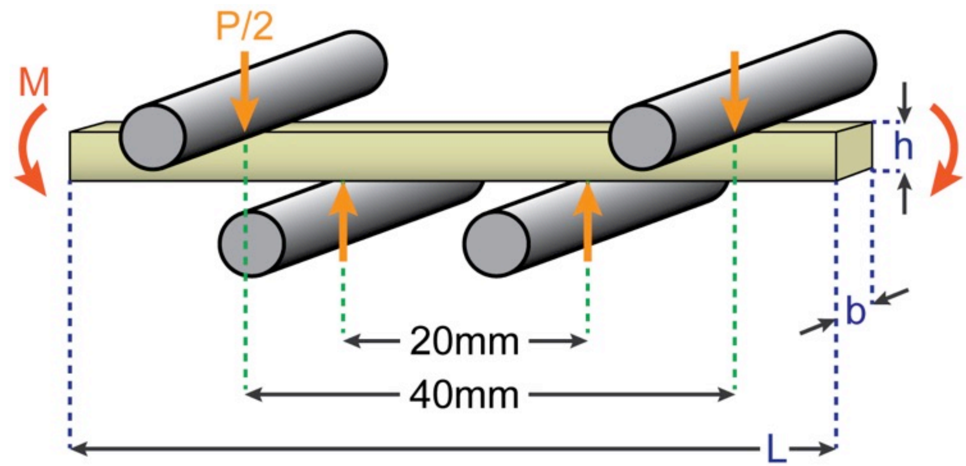
  15 YbS2 and and 15 Si

  @ 50 x 5 x 4mm
- Test density, size, elastic modulus for all specimens
- Perform mechanical tests

4pt flexure- isothermal & thermal gradient

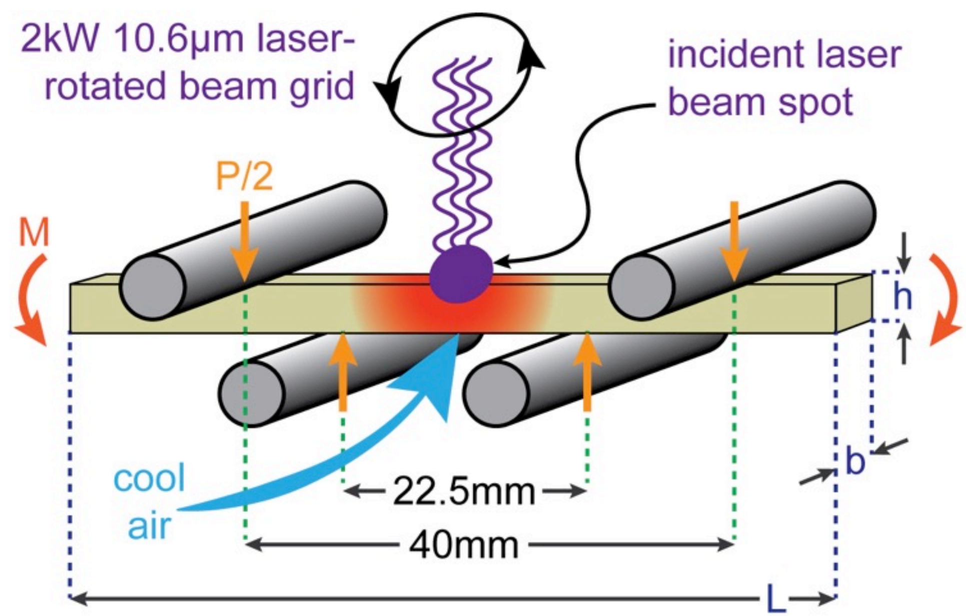
Convenient for determining many mechanical properties

Low temperature configuration (ASTM C1161)



High temperature configuration (ASTM C1211)

Laser/cooling air induced thermal gradient configuration



Creep Equations and Testing

Primary

$d\varepsilon/dt = A * \exp(-E_a/RT) * \sigma^n * t^{-s}$

key parameters $\mathbf{E_a}$, **n**, **s**

Secondary

$d\varepsilon/dt = C * \exp(-E_a/RT) * \sigma^n$

key parameters $\mathbf{E_a}$, **n**

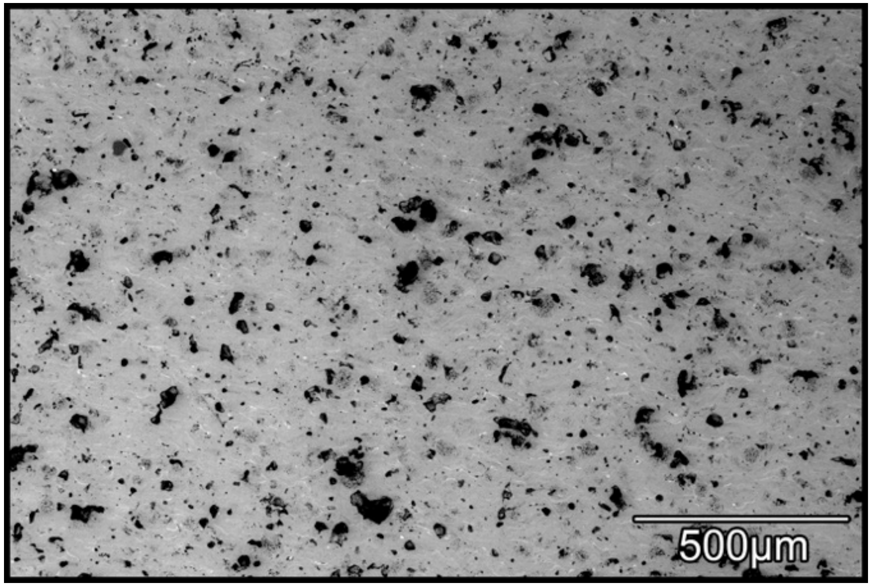
Data recorded in creep test is actuator or mid-point displacement, load, and time. Models are used to calculate strain (see Hollenberg et. al. 1970).

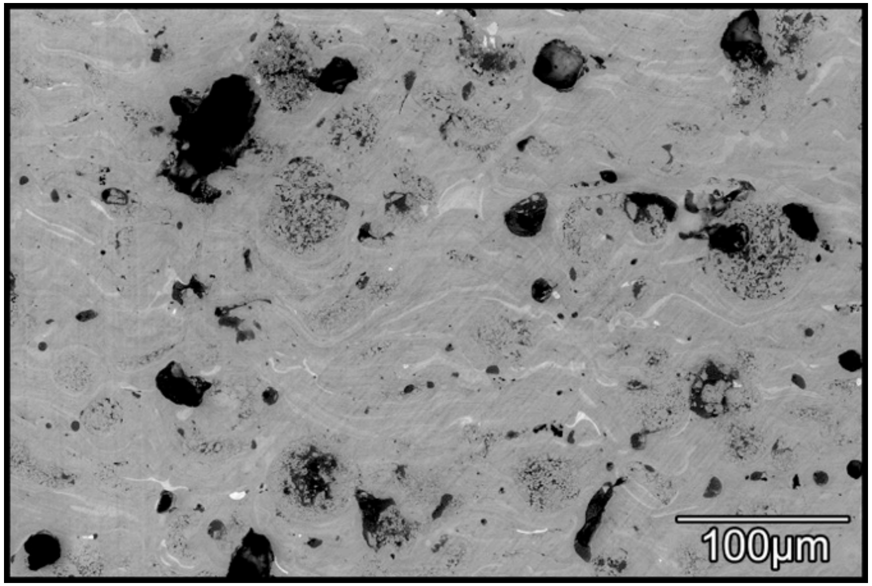
For low n (~1), elastic strains from Euler-Bernoulli beam theory happen to be accurate.

APS $Yb_2Si_2O_7$ Properties

Elastic Modulus, Flexure Strength, Toughness, Isothermal Creep, Laser Thermal Gradient Creep

$Yb_2Si_2O_7$ Microstructure





APS structure crack-free and 90-91% dense (archimedes density and image analysis)

Pores loosely spherical

Some Si loss occurred during APS processing (quantity still undetermined, pending analysis)

$Yb_2Si_2O_7$ Properties

- Elastic Modulus- impulse excitation E = 33.4 GPa, σ = 2.53
  13 specimens @ 25°C

(~170-180GPa bulk: X-ray, nanoindentation)

- Low Temperature K_{IC} = 0.925 MPa*$m^{0.5}$, σ = 0.05
  3 specimens @ 25°C

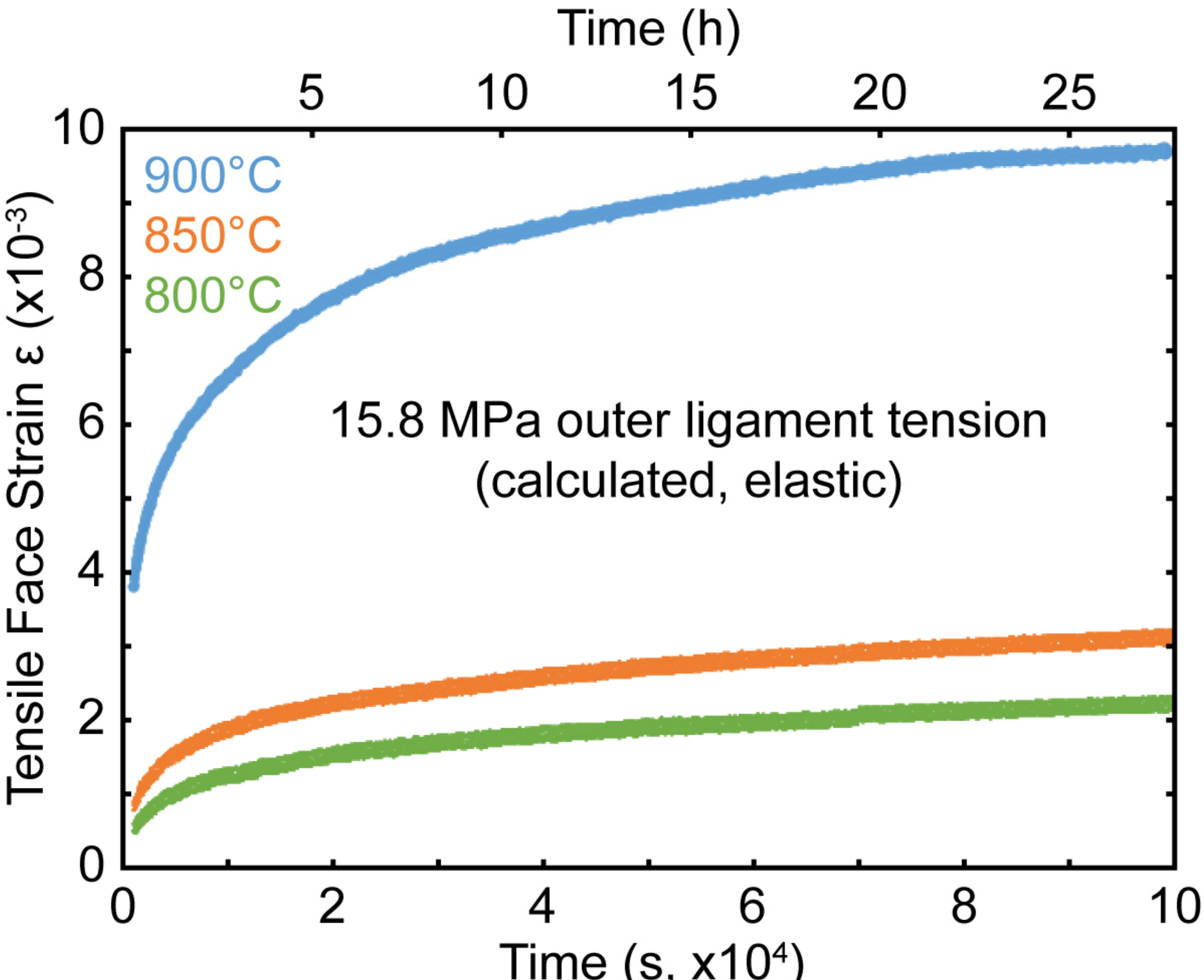
(dense material 2 MPa*$m^{0.5}$)

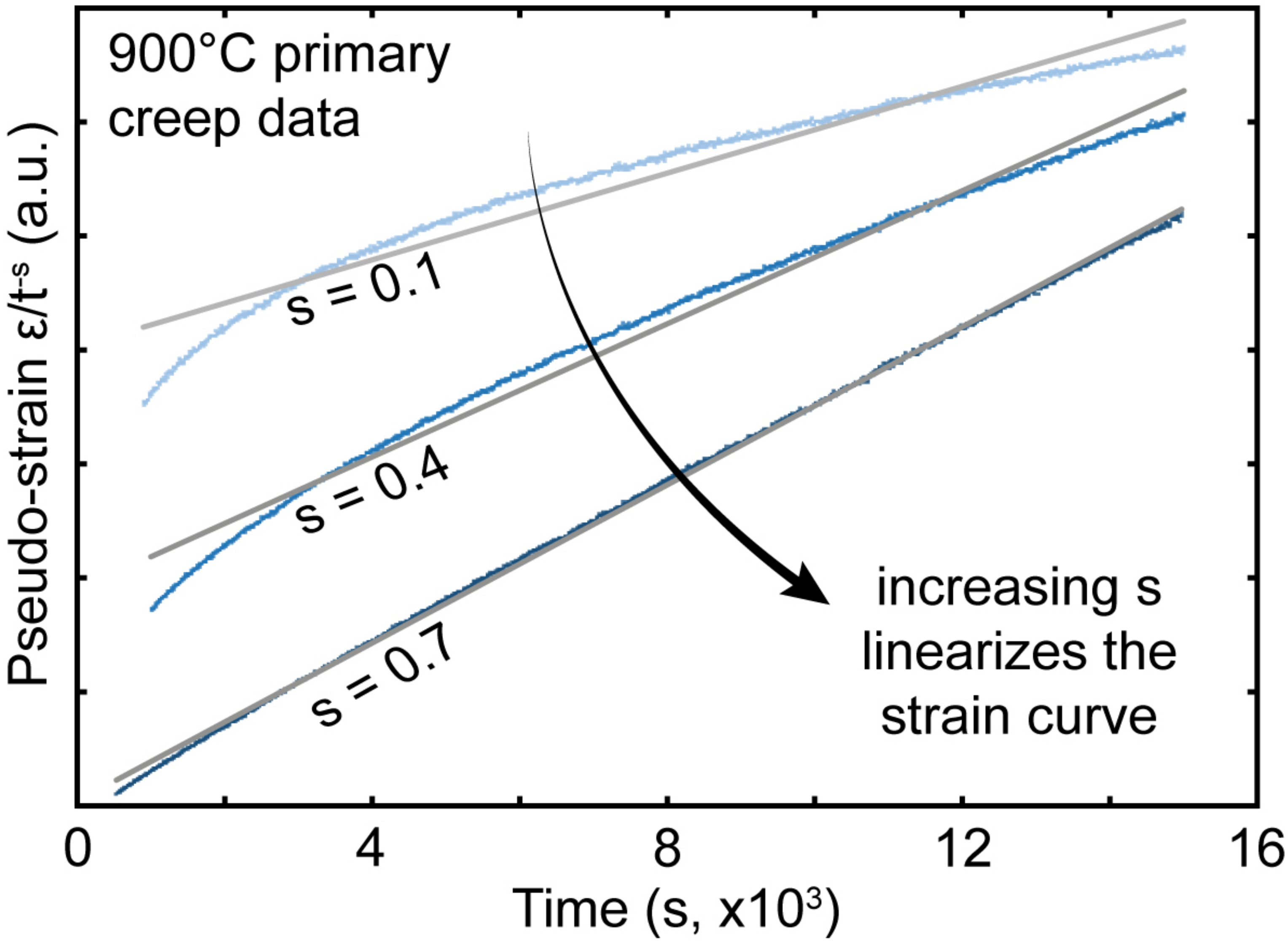
- Low Temperature Flexure Strength = 19.7 MPa, σ = 0.62
  4 specimens @ 25°C

(dense & stoichiometric: ~100MPa $Yb_2Si_2O_7$, ~15MPa Yb_2SiO_5)

- High Temperature Flexure Strength = 24.3 MPa, σ = 0.42
  2 specimens @ 900°C

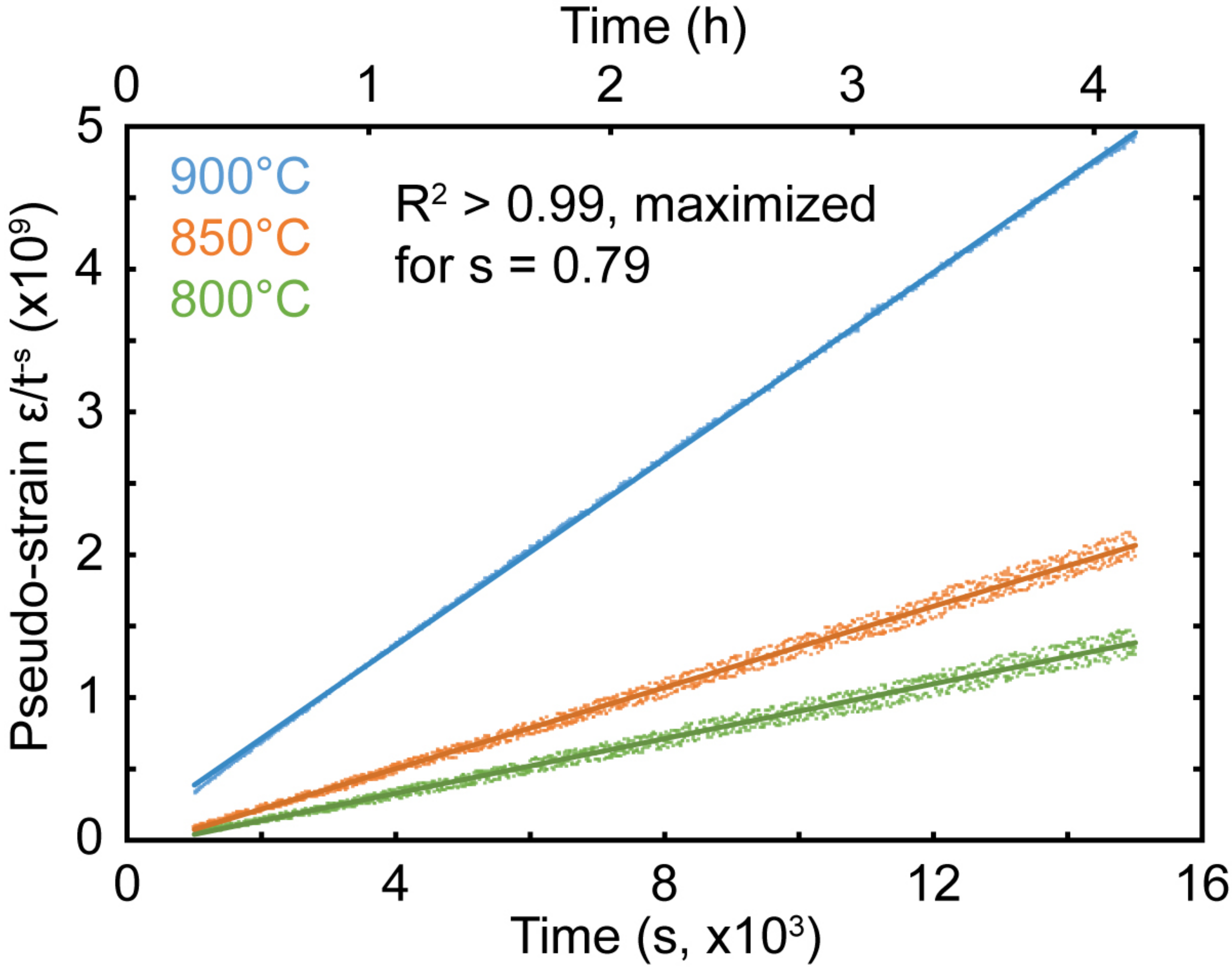
Isothermal Creep Behavior





900°C primary creep data
s = 0.1
s = 0.4
s = 0.7
increasing s linearizes the strain curve
Pseudo-strain ε/t^-s (a.u.)
0
4
8
12
16
Time (s, x10^3)

Primary creep s parameter determination



Isothermal Creep Rates and E_a

Isothermal Secondary Creep Strain Rates @ 15.8 MPa:

$d\varepsilon/dt = 3.39 \times 10^{-9}$ @ 800°C

$d\varepsilon/dt = 5.63 \times 10^{-9}$ @ 850°C

$d\varepsilon/dt = 1.23 \times 10^{-8}$ @ 900°C

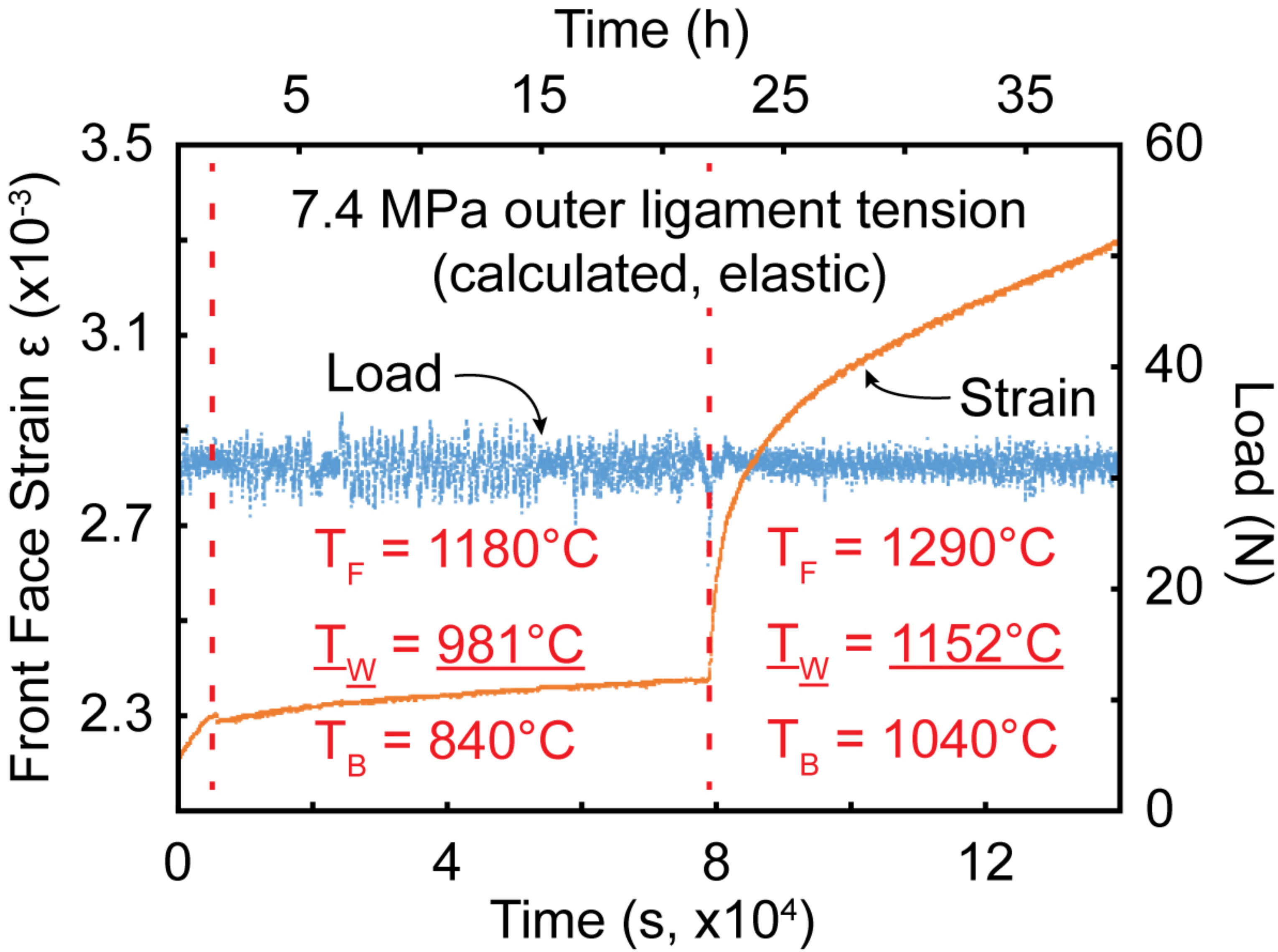
$E_{a,secondary} = 134.7$ kJ/mol with $R^2 = 0.98$

$E_{a,primary} = 127.2$ kJ/mol with $R^2 = 0.94$

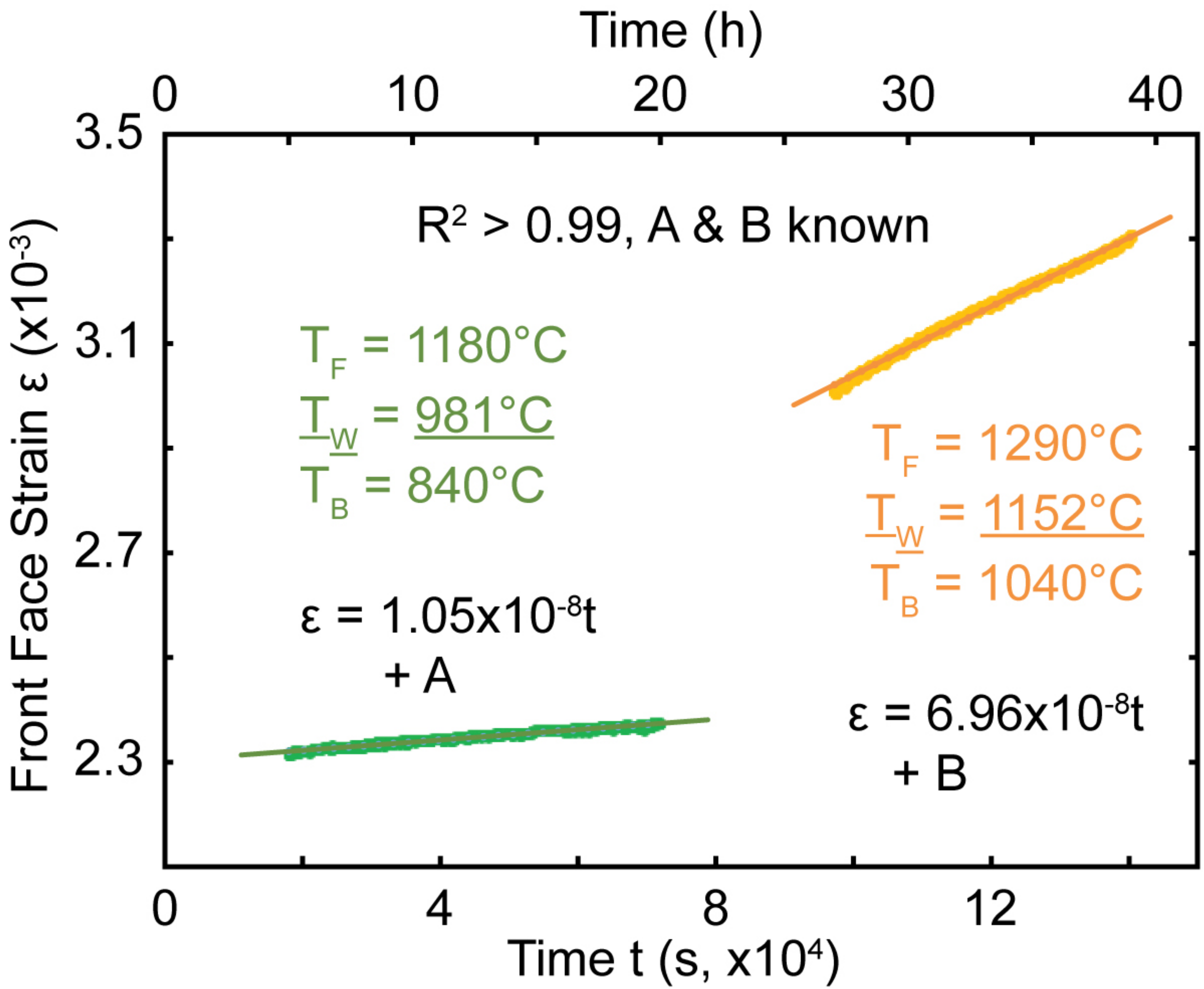
For reference (see Zhu et. Al. 1999):

E_a for 10% porous APS YSZ = 105 kJ/mol

Laser Thermal Gradient Creep Behavior



Thermal Gradient Creep Behavior (T)



Creep Activation Energies

Isothermal Secondary Creep Activation Energy

E_a = 134.7 kJ/mol @ 15.8 Mpa

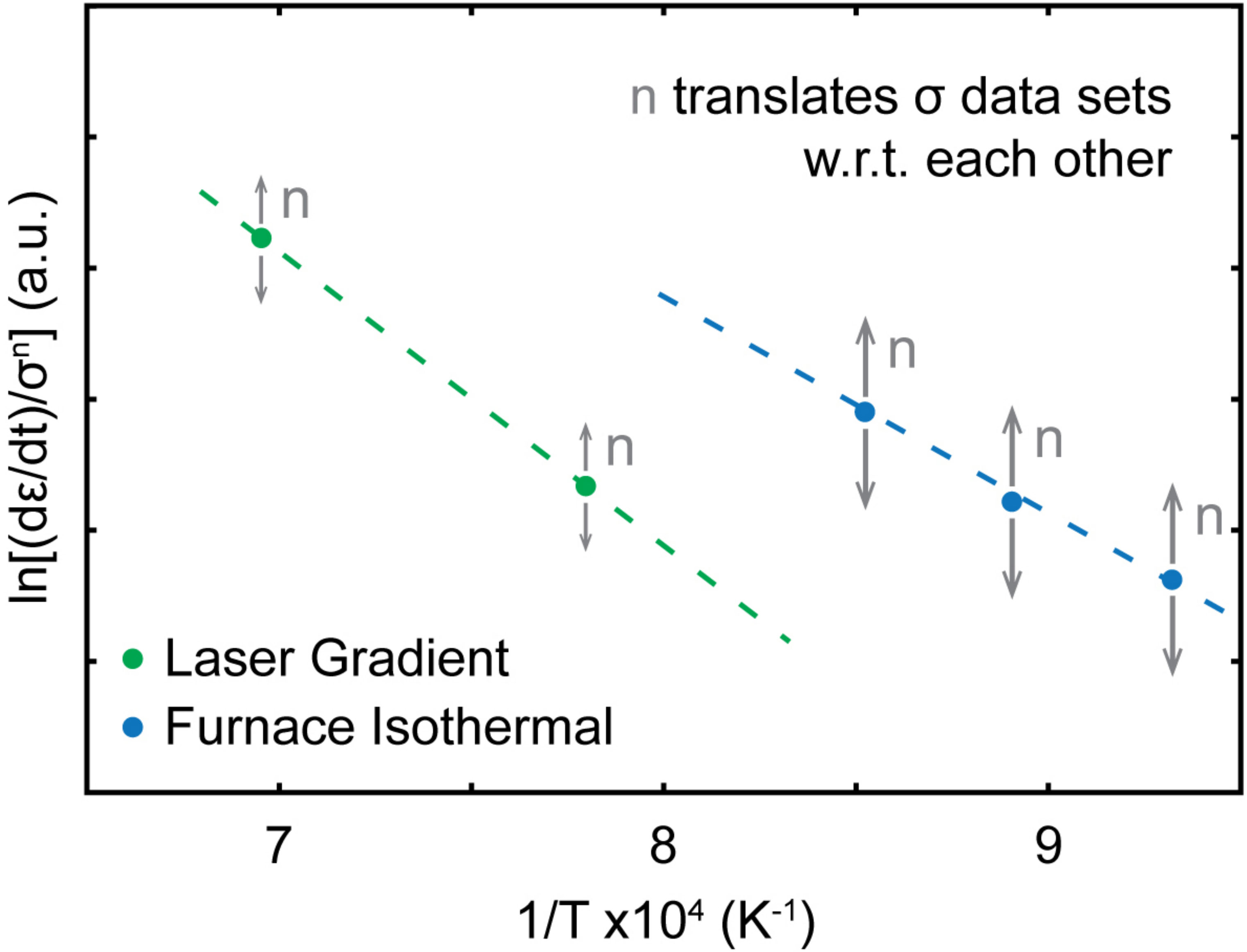
Laser Gradient Secondary Creep Activation Energy

E_a = 165.4 kJ/mol @ 7.4 Mpa

Data at multiple stresses allows rough approximation of creep n parameter assuming same creep mechanism (same E_a).

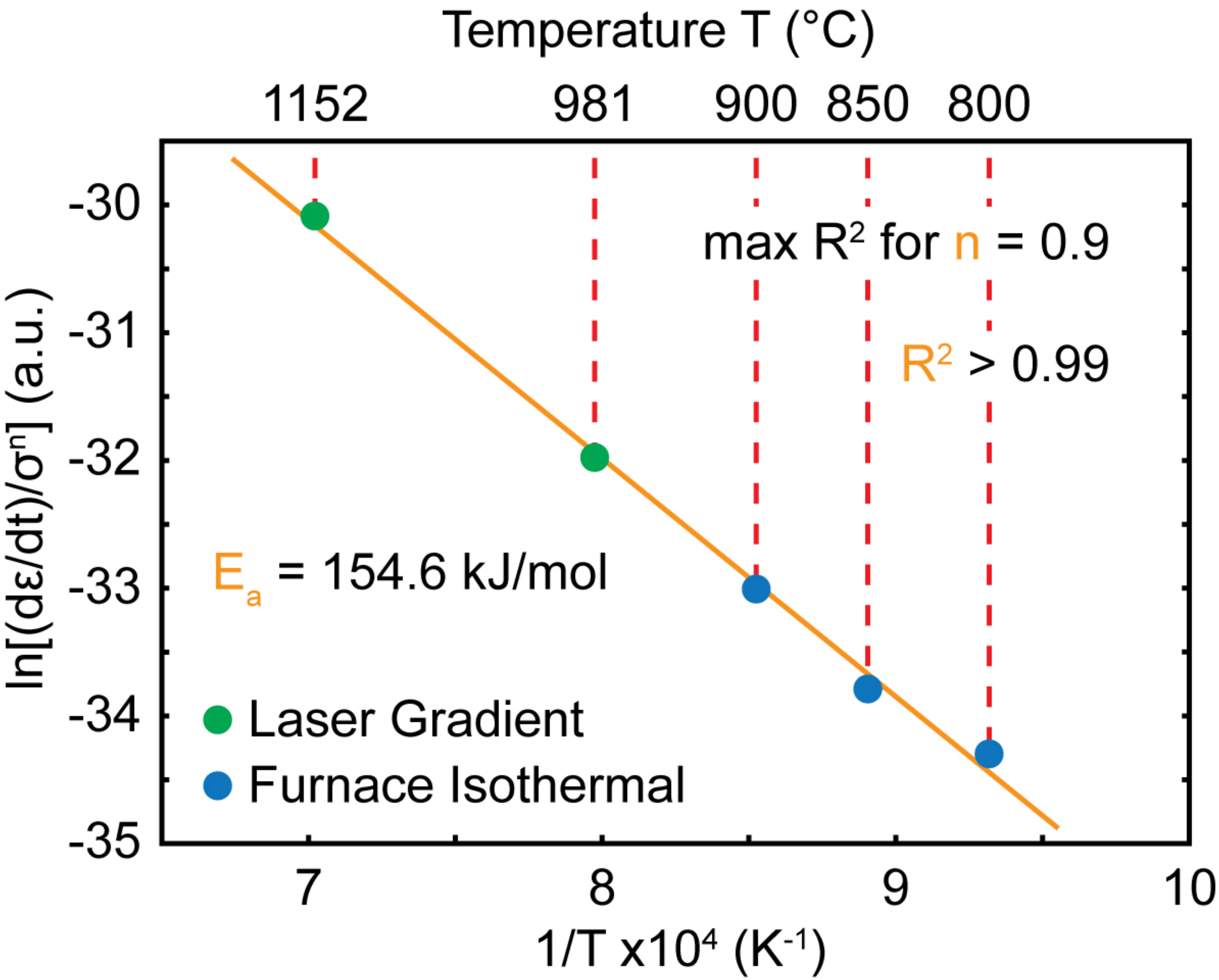
n will be a rough estimate!!! More detailed determination of n with isothermal data in progress.

Effect of n



n translates σ data sets
w.r.t. each other
n
n
n
n
n
Laser Gradient
Furnace Isothermal
7
8
9
1/T x10⁴ (K⁻¹)
ln[(dε/dt)/σⁿ] (a.u.)

n and E_a determinations



APS $Yb_2Si_2O_7$ Properties Summary

E = 33.4 GPa (~180GPa bulk- X-ray, nanoindentation)

K_{IC} = 0.925 MPa*$m^{0.5}$

Low T Flexure Strength = 19.7 Mpa (~100 MPa for dense, stoichiometric $Yb_2Si_2O_7$, ~15 MPa for Yb_2SiO_5)

High T Flexure Strength = 24.3 Mpa

dε/dt = 3.39 $x10^{-9}$ @ 800°C (isothermal)

dε/dt = 6.96 $x10^{-8}$ @ 1152°C (laser gradient)

s = 0.78 (comparable to APS YSZ)

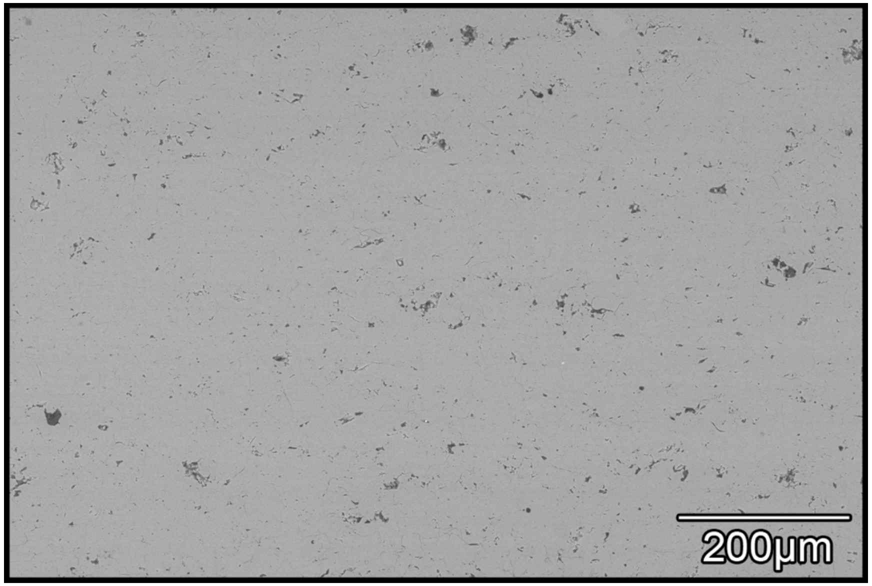
n = ~0.9 (n = 1 for diffusional)

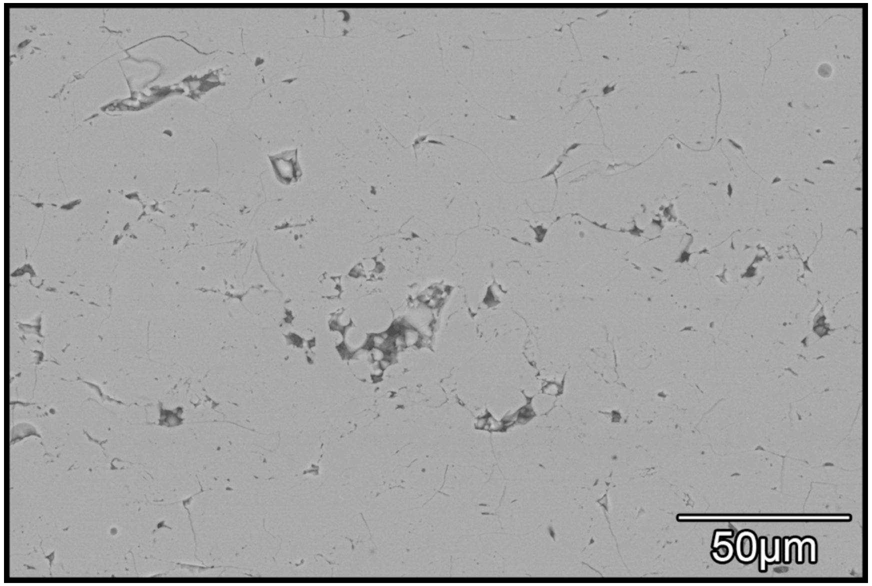
E_a = 135-165 kJ/mol

APS Silicon Properties

Elastic Modulus, Flexure Strength, Toughness, Laser Thermal Gradient Creep, Interface Toughness

Si Microstructure





Plasma sprayed structure is crack-free and 95+% dense (archimedes density and image analysis)

APS Si Properties (uncoated)

- Elastic Modulus- impulse excitation E = 73.0 GPa, $\sigma = 2.12$
  9 specimens @ 25°C

(E = 163 GPa for pure, dense polycrystal)[a]

- Low Temperature $K_{IC} = 1.54$ MPa*$m^{0.5}$, $\sigma = 0.05$
  4 specimens @ 25°C

(0.8-0.9 MPa*$m^{0.5}$)[a]

- Low Temperature Flexure Strength = 76.6 MPa, $\sigma = 1.32$
  5 specimens @ 25°C

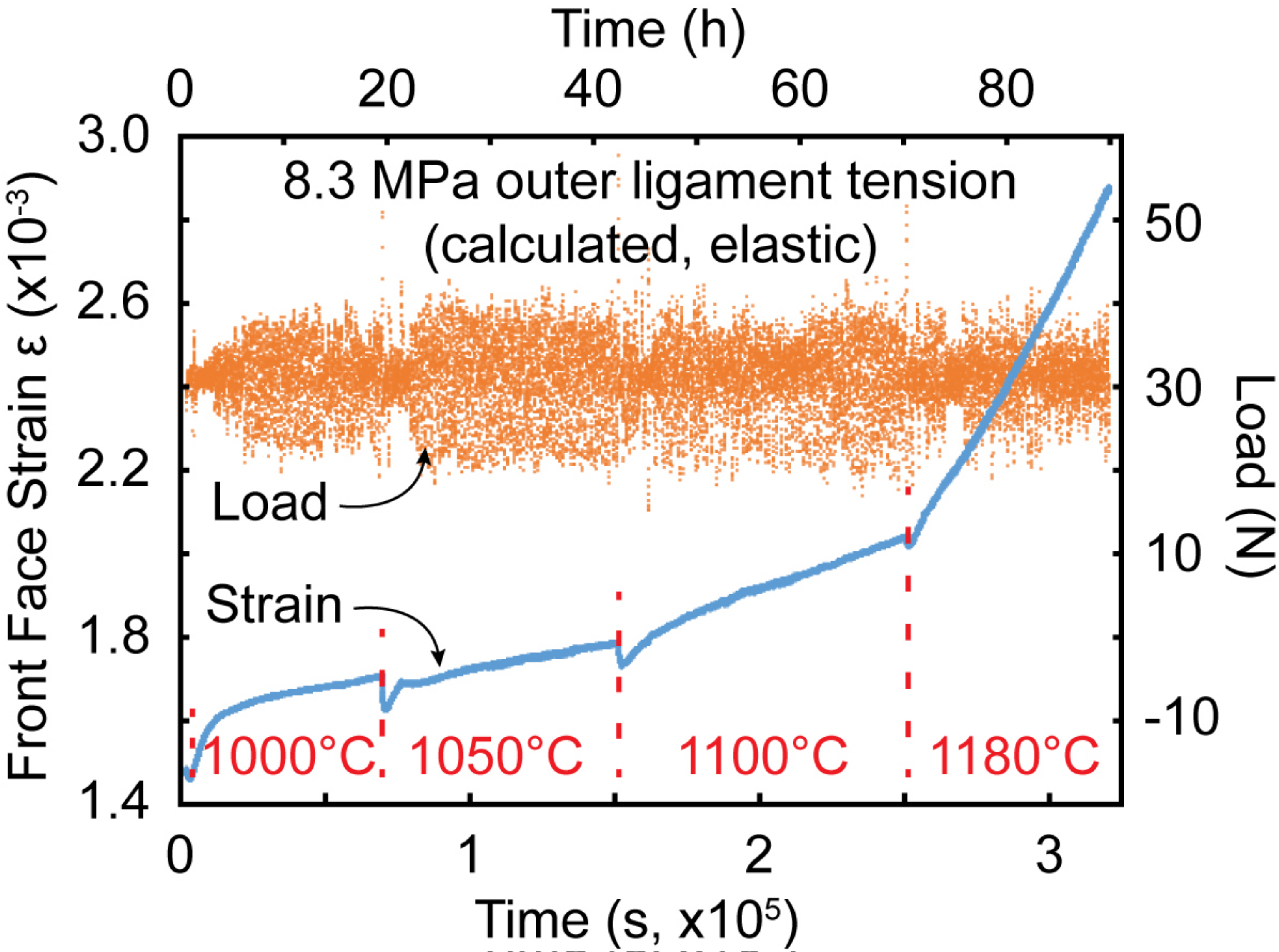
(260 MPa polished polycrystal, very high scatter)[a]

- High Temperature Flexure Strength = 186.8 MPa, $\sigma = 24.2$
  2 specimens @ 900°C

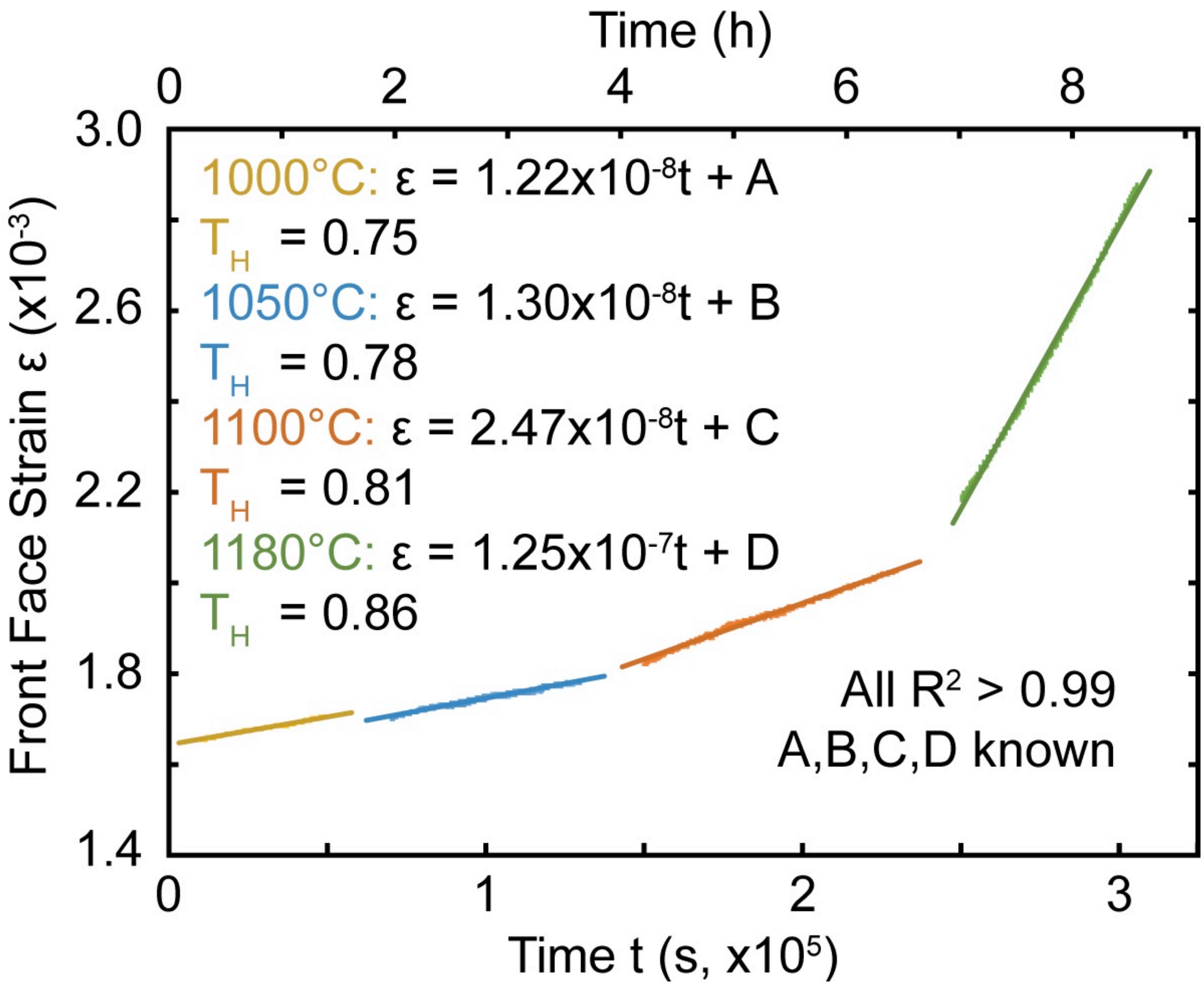
(500 MPa polished polycrystal, very high scatter)[a]

[a]Hull, 1999

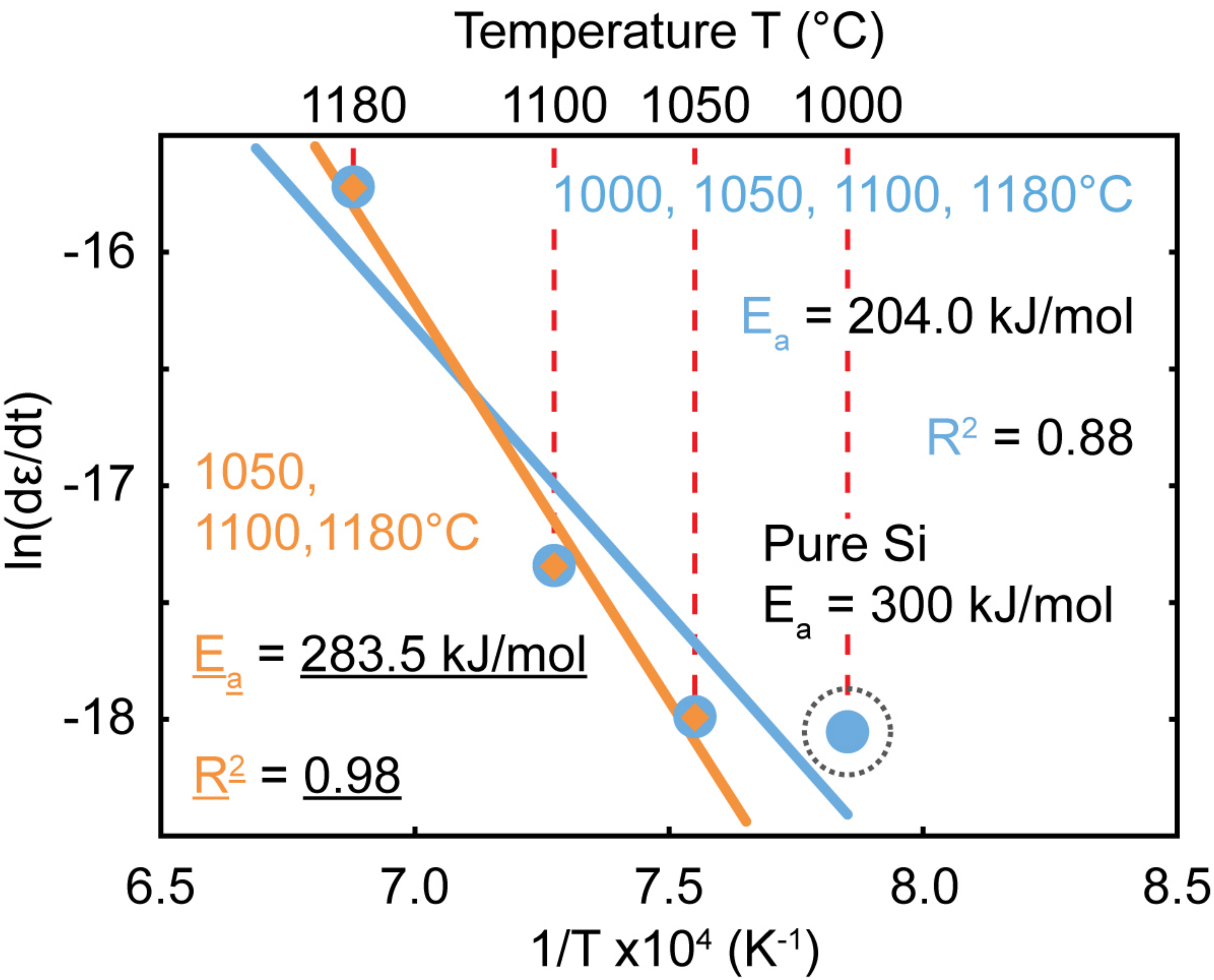
4 T YbS2-coated laser thermal gradient creep



Gradient Creep Behavior at Different T



E_a calculation for APS Si



Laser Gradient Creep Rates and E_a

Isothermal Secondary Creep Strain Rates (mechanical affect of 200µm YbS2 not considered):

$d\varepsilon/dt = 1.22 \times 10^{-8}$ @ 1000°C

$d\varepsilon/dt = 1.29 \times 10^{-8}$ @ 1050°C

$d\varepsilon/dt = 2.47 \times 10^{-8}$ @ 1100°C

$d\varepsilon/dt = 1.25 \times 10^{-7}$ @ 1180°C

**1000°C data may be suspect/ different creep regime*

Isothermal Secondary Creep Activation Energy

E_a = 283.5 kJ/mol with $R^2 > 0.98$ (bulk 300 kJ/mol) (1050, 1100, 1180°C data)

APS Si Properties Summary

E = 73.0 GPa (163 GPa bulk)

K_{IC} = 1.54 MPa*m$^{0.5}$ (0.8-0.9 MPa*m$^{0.5}$)

Low T Flexure Strength = 76.6 Mpa (~260MPa bulk)

High T Flexure Strength = 186.6 Mpa (~500MPa bulk)

dε/dt = 1.29 x10^{-8} @ 1050°C (laser gradient)

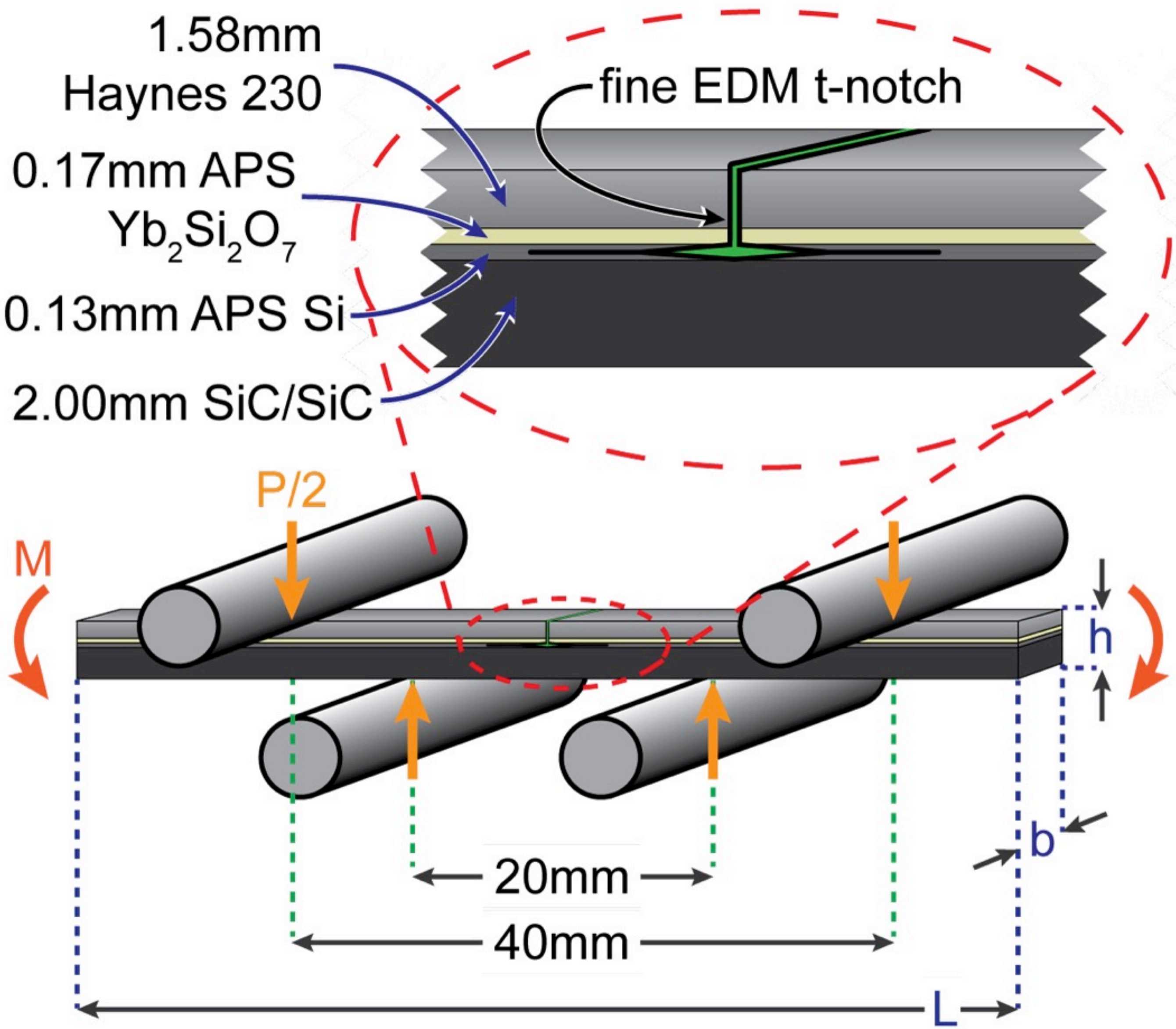
dε/dt = 1.25 x10^{-7} @ 1180°C (laser gradient)

E_a = 283.5 kJ/mol (300 kJ/mol bulk)

*Increase in K_{IC} may be due to crack blunting, splat boundary SiO_2, reduced modulus, etc.

Interlaminar (interfacial) Toughness

4-point bend testing of cracked specimens



EBC Interfacial/ interlaminar toughness studies:

4-point bending t-notched configuration

FEM modeled fracture solutions

Stress Intensity Factors

$$F_I(a) = K_I\, BW^{3/2}/(P(S_o\text{-}S_i))$$

$$F_{II}(a) = K_{II}\, BW^{3/2}/(P(S_o\text{-}S_i))$$

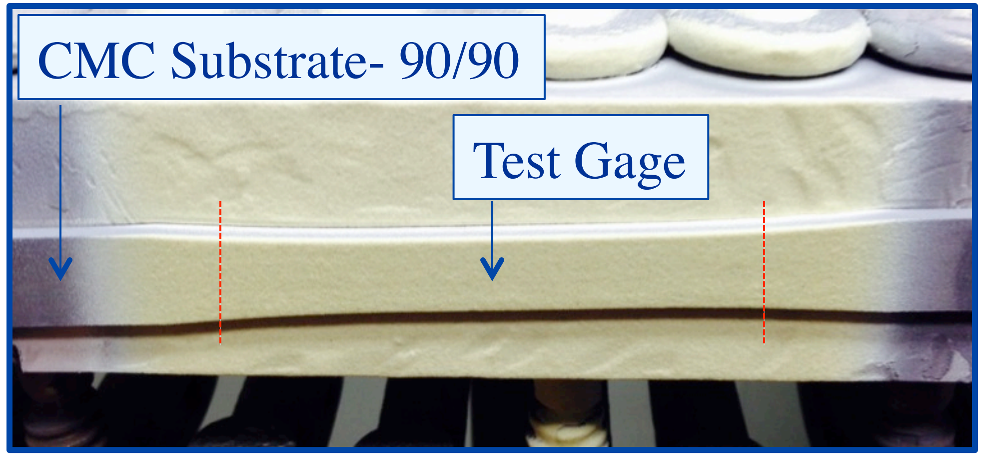
Strain Energy Release Rate

$$G(a) = K_I^{\,2}(1\text{-}\nu^2)/E + K_{II}^{\,2}(1\text{-}\nu^2)/E$$

$$G_N = GP(S_o\text{-}S_i)/(BW^2)$$

Mode Mixity Angle

$$\phi = atan(K_{II}/K_I)$$

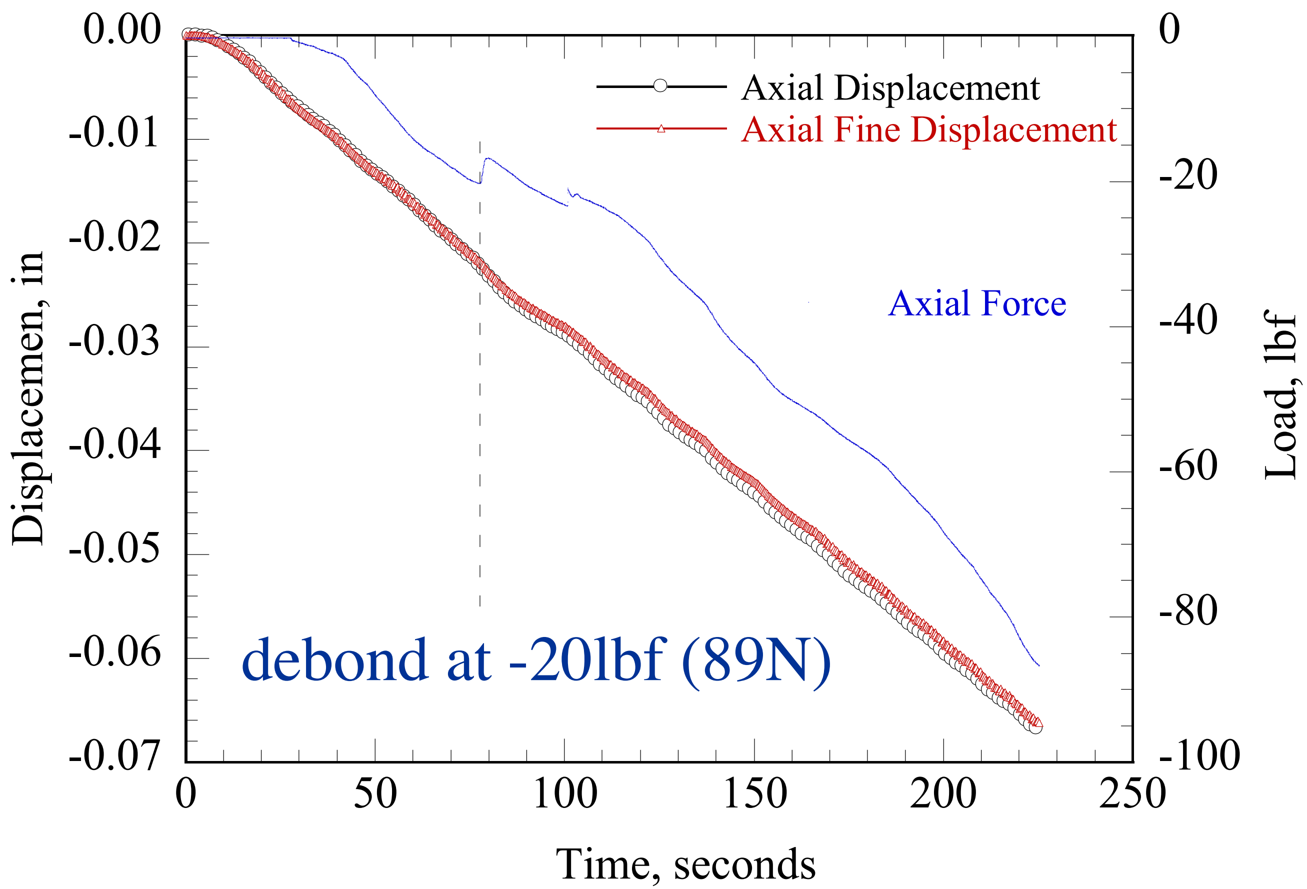


Air Plasma Sprayed CMC prior to stiffener bonding & test form machining

a/2 mm	a/Si	KI-norm	KII-Norm	Angle, deg	G-Norm
2	0.1	1.07	0.59	29.10	4.89E-05

Data used in calculations

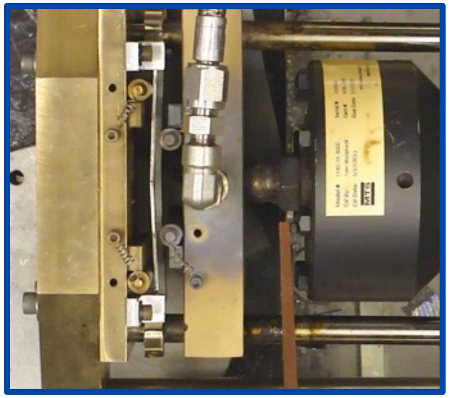
Example Test on T-cracked Specimen crack appears to kink into Si-SiC interface



At -20 lbf (89 N)
$K_I = 1.576\ MPa*m^{0.5}$
$K_{II} = 0.869\ MPa*m^{0.5}$

At -22 lbf (98 N)
$K_I = 1.733\ MPa*m^{0.5}$
$K_{II} = 0.956\ MPa*m^{0.5}$

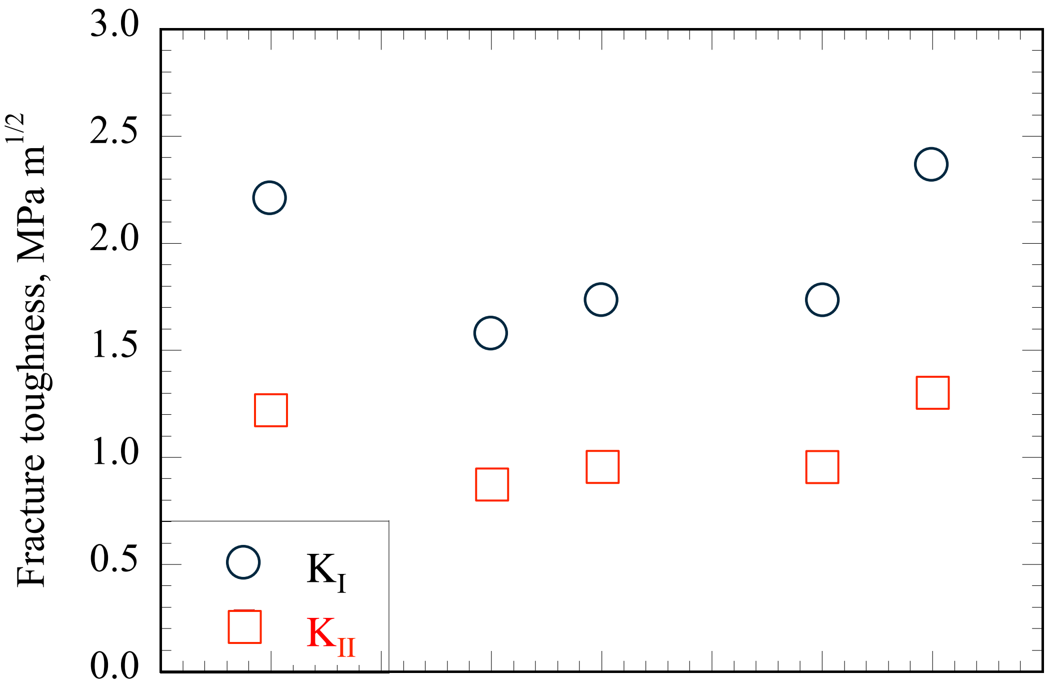
Debonded in load frame



After test

EBC Interlaminar Toughness Results

Measured K_I = 1.92 MPa*$m^{0.5}$ and K_{II} = 1.06 MPa*$m^{0.5}$ at φ = 29.10° and critical load (i.e. G_C achieved)- calculated using FEM solutions presented.



	K1	KII
Min	1.58	0.87
Max	2.36	1.30
Mean	1.92	1.06
Std Dev	0.34	0.188

Conclusions

1. Elastic Moduli and Flexure Strength of APS EBC materials are low, but comparable to data published for APS TBC materials
2. Fracture toughness of APS materials is comparable to toughness of bulk materials
3. Creep rates of EBC materials appear high and activation energies for YbS2 are low- creep is likely a surface diffusion based process
4. Interlaminar toughness measurements are possible using a stiffener-modified 4-point bend test; Si-SiC interface toughnesses have been measured and appear comparable to Si

Acknowledgements and Questions

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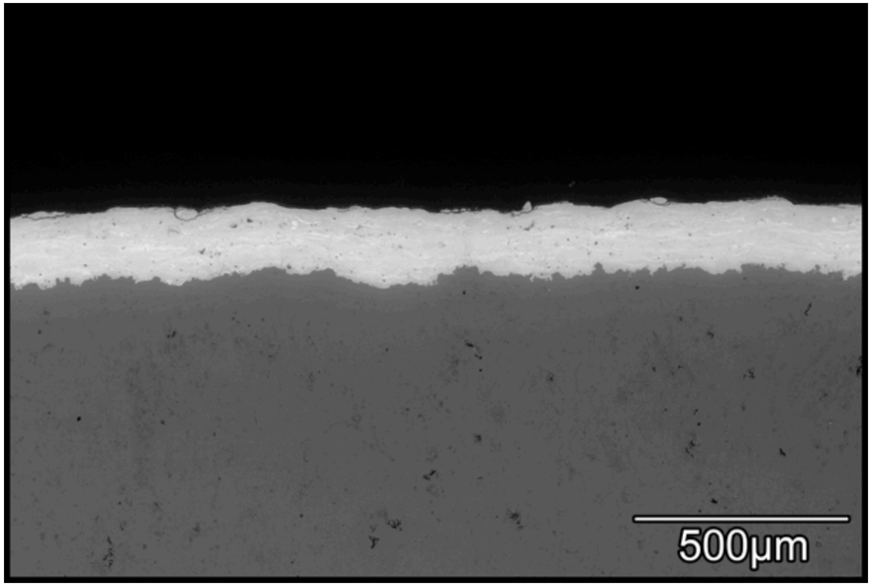
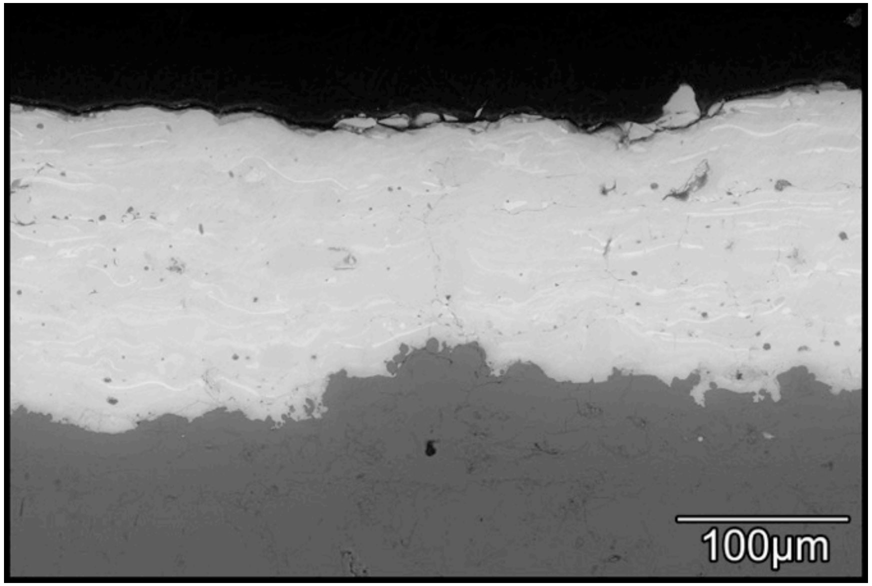
Backup Slides

(and Appendix-type data)

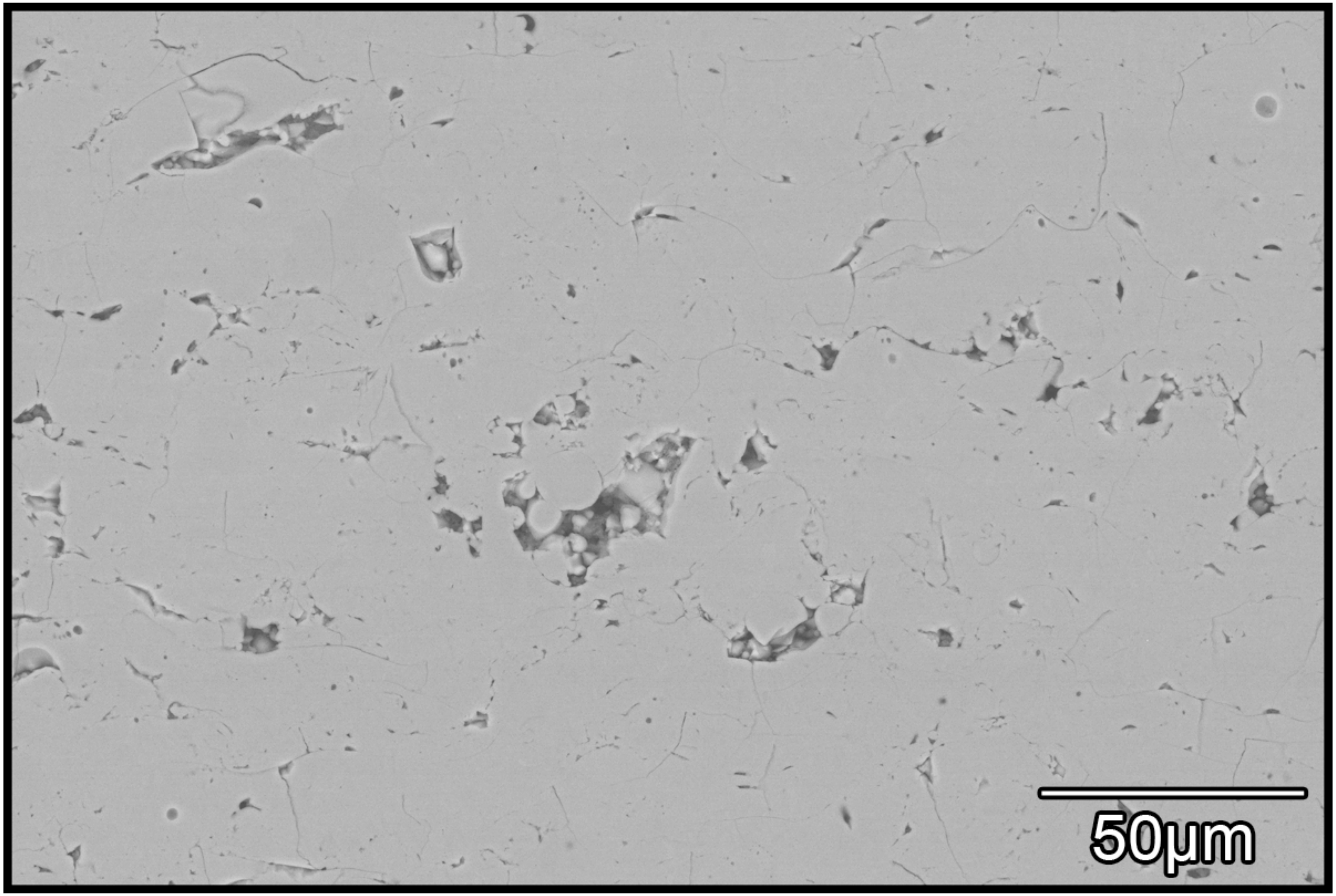
Si Microstructure

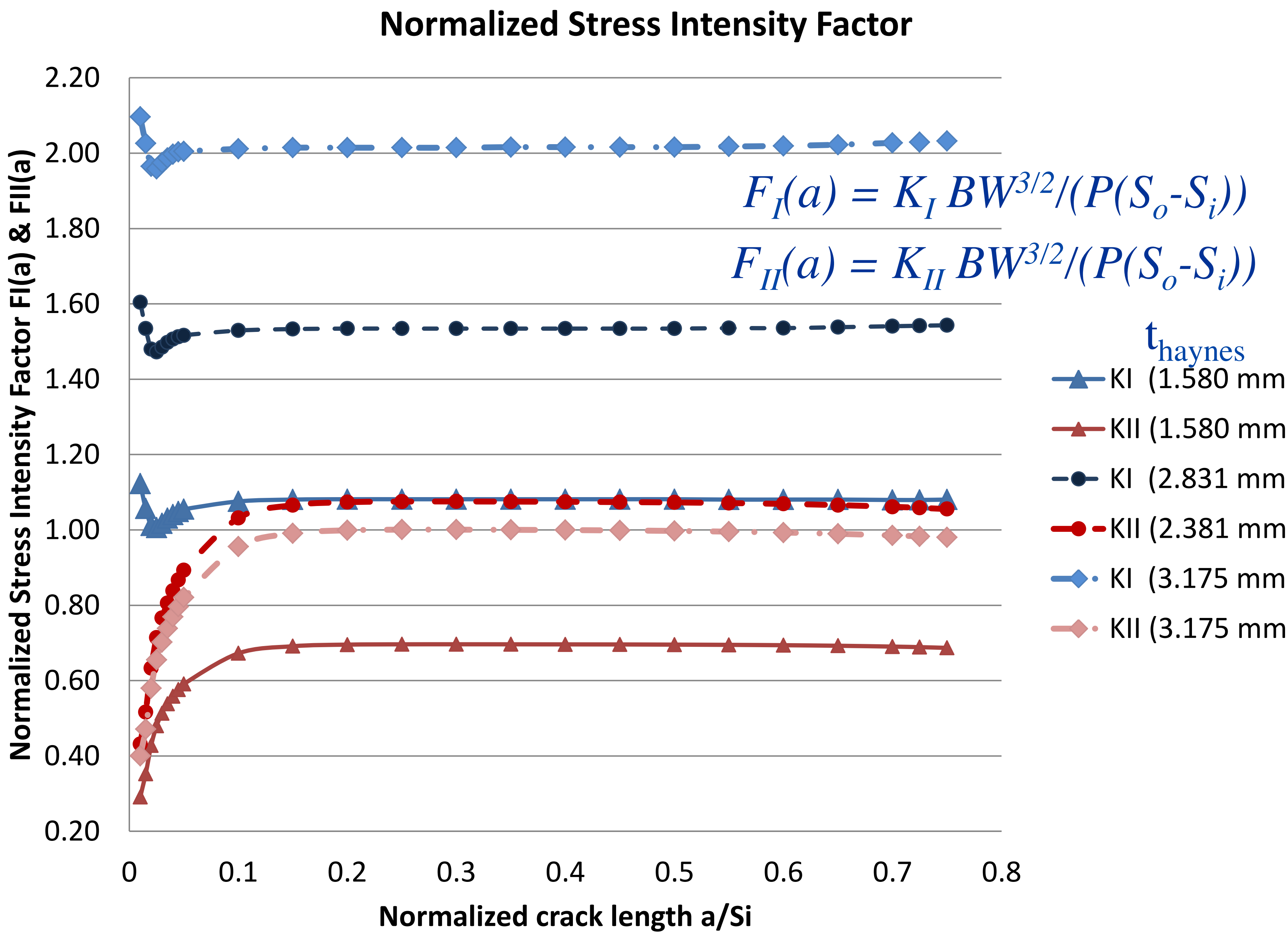


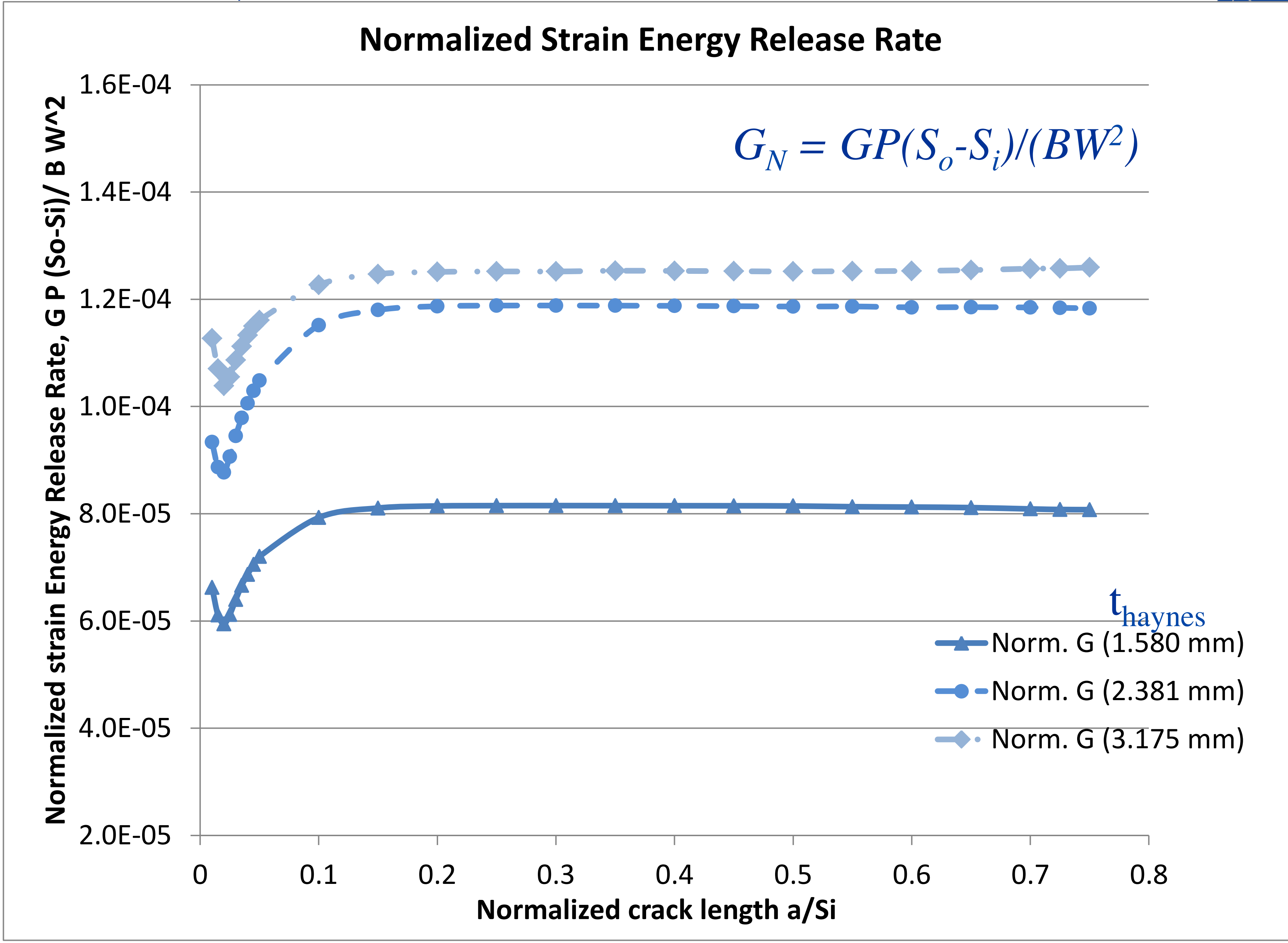


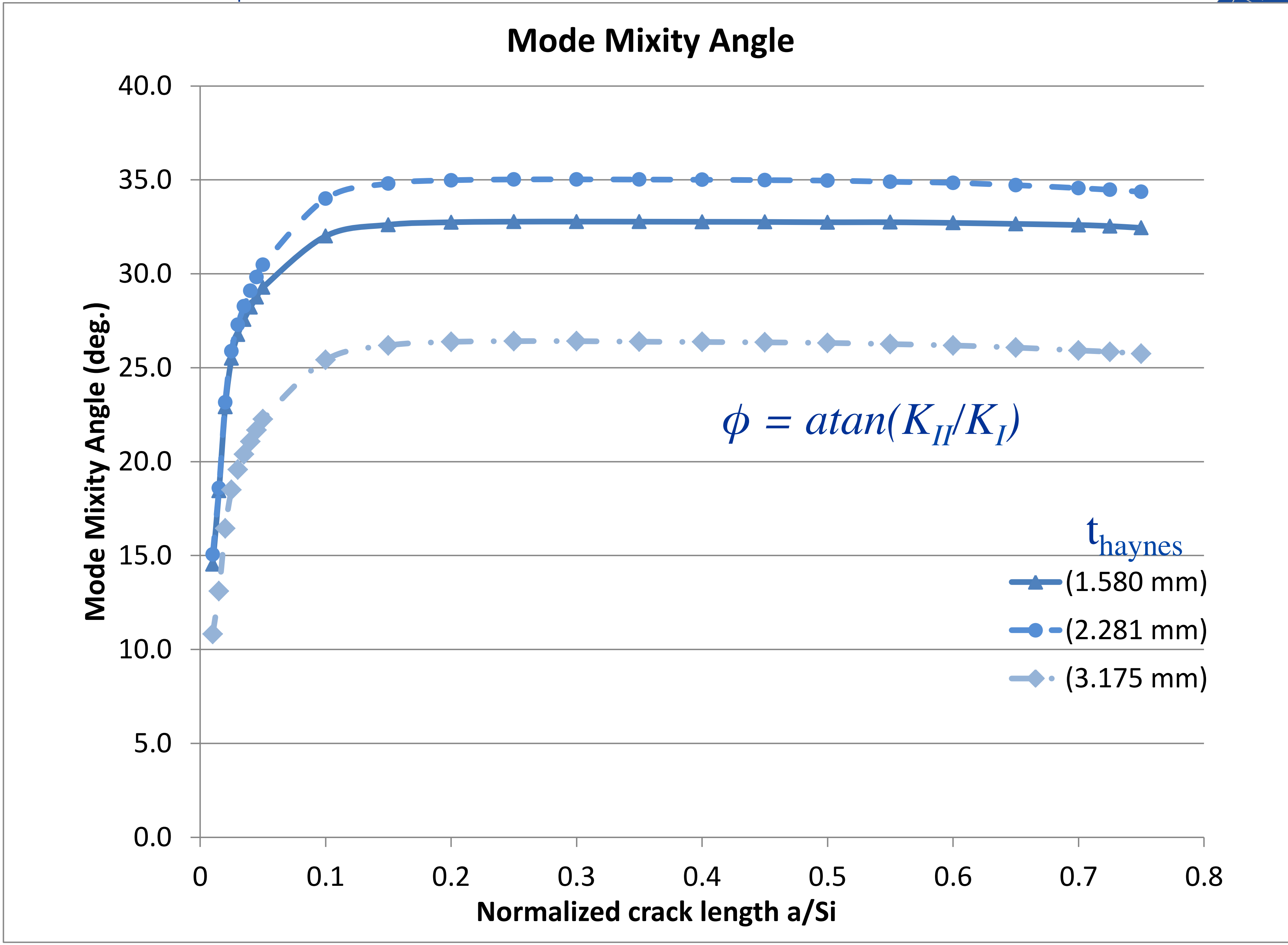
Plasma sprayed structure is crack-free and 94-95% dense (archimedes density and image analysis)

Higher Magnification APS Si




Normalized Stress Intensity Factor
$F_I(a) = K_I\, BW^{3/2}/(P(S_o\text{-}S_i))$
$F_{II}(a) = K_{II}\, BW^{3/2}/(P(S_o\text{-}S_i))$
t_{haynes}
KI (1.580 mm)
KII (1.580 mm)
KI (2.831 mm)
KII (2.381 mm)
KI (3.175 mm)
KII (3.175 mm)
Normalized Stress Intensity Factor FI(a) & FII(a)
Normalized crack length a/Si
2.20
2.00
1.80
1.60
1.40
1.20
1.00
0.80
0.60
0.40
0.20
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8

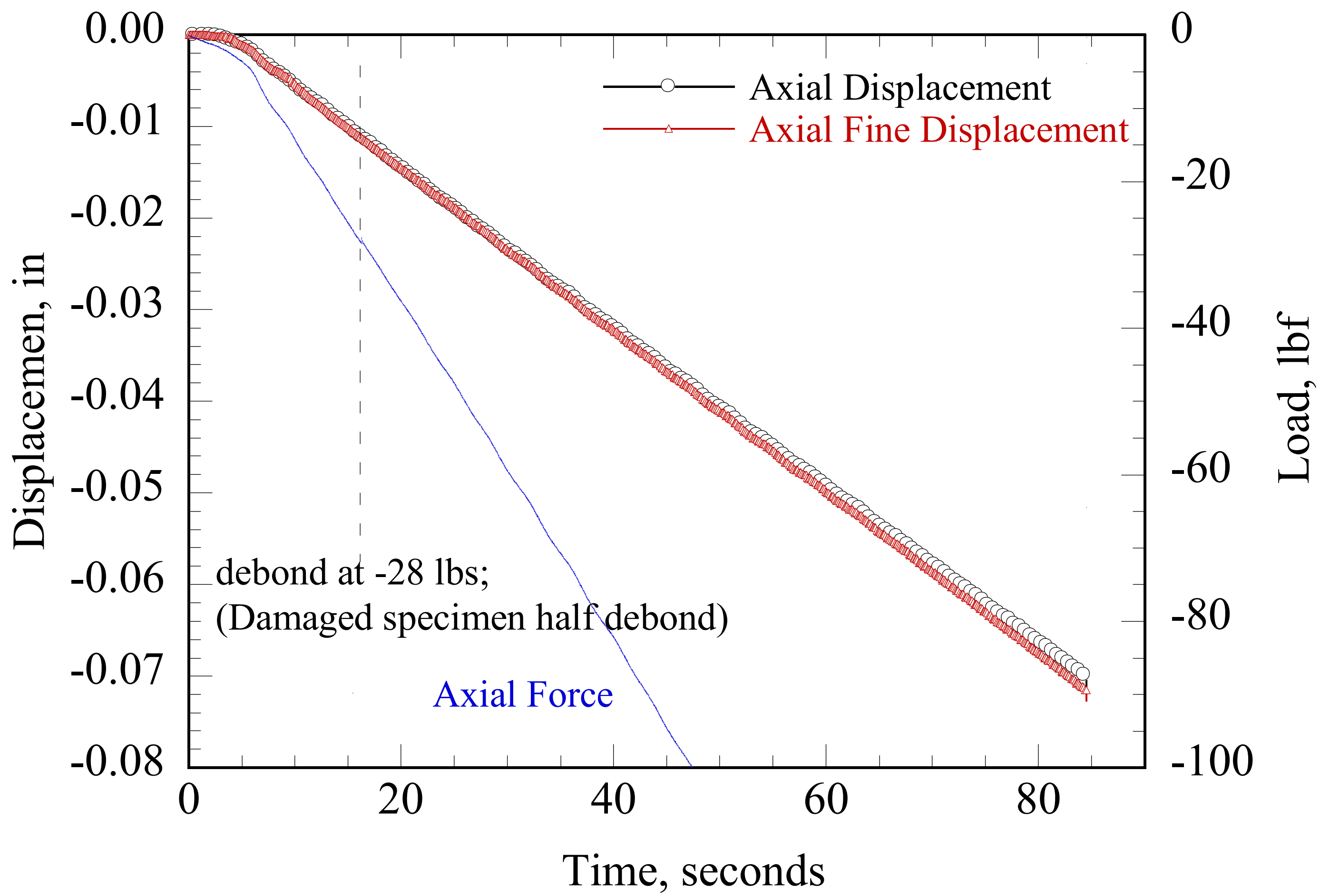

Normalized Strain Energy Release Rate
$G_N = GP(S_o\text{-}S_i)/(BW^2)$
Normalized strain Energy Release Rate, G P (So-Si)/ B W^2
1.6E-04
1.4E-04
1.2E-04
1.0E-04
8.0E-05
6.0E-05
4.0E-05
2.0E-05
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
Normalized crack length a/Si
t_{haynes}
Norm. G (1.580 mm)
Norm. G (2.381 mm)
Norm. G (3.175 mm)



Mode Mixity Angle
40.0
35.0
30.0
25.0
20.0
15.0
10.0
5.0
0.0
Mode Mixity Angle (deg.)
0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
Normalized crack length a/Si
$\phi = atan(K_{II}/K_I)$
t_{haynes}
(1.580 mm)
(2.281 mm)
(3.175 mm)

EBC Interlaminar Strength Test - 114-93-7-#13

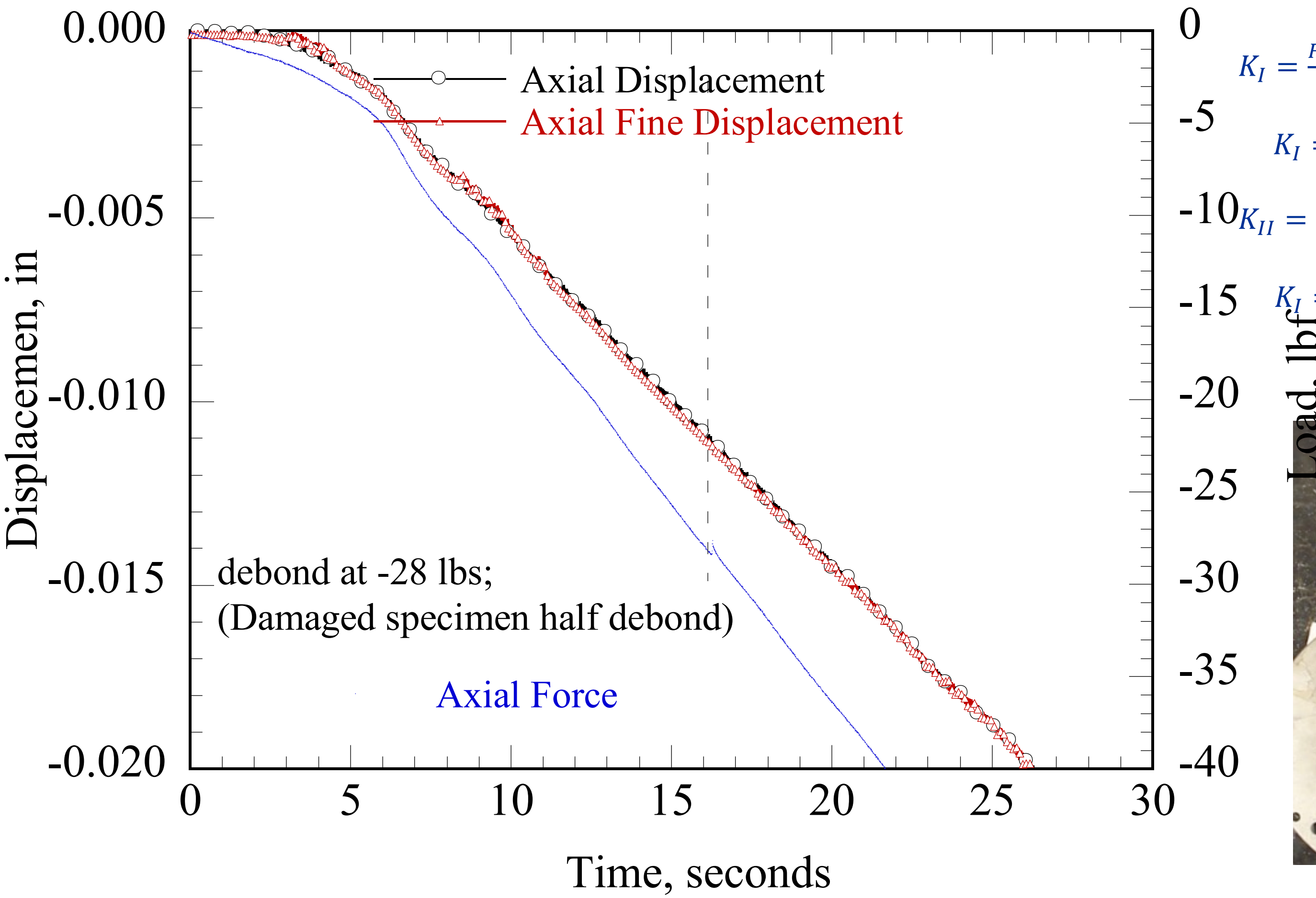
- 114-93-7-#13 specimen, failure load at -28 lbf



After debond

EBC Interlaminar Strength Test - 114-93-7-#13 Cont'd

- 114-93-7-#13 specimen, failure load at -28 lbf



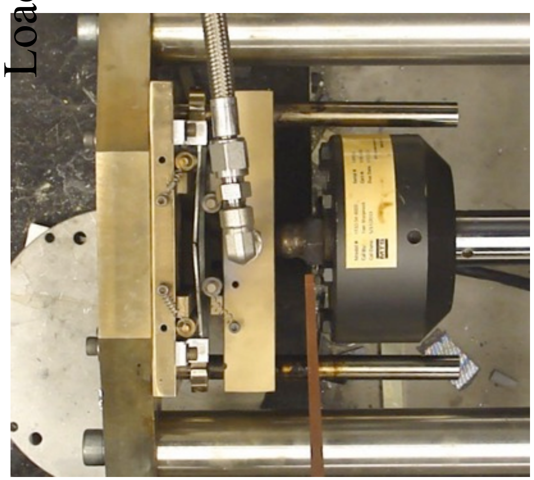
At -28 lbf (124.578N)

$$K_I = \frac{F_I(a)P\,(S_o - S_i)}{B\,W^{3/2}} = \frac{1.07*124.578*40*10^{-3}}{(10*10^{-3})*(3.88*10^{\wedge}-3)^{3/2}}$$

$$K_I = 2.206\ MPa * m^{0.5}$$

$$K_{II} = \frac{F_{II}(a)P\,(S_o - S_i)}{B\,W^{3/2}} = \frac{0.59*124.578*40*10^{-3}}{(10*10^{-3})*(3.88*10^{\wedge}-3)^{3/2}}$$

$$K_I = 1.216\ MPa * m^{0.5}$$

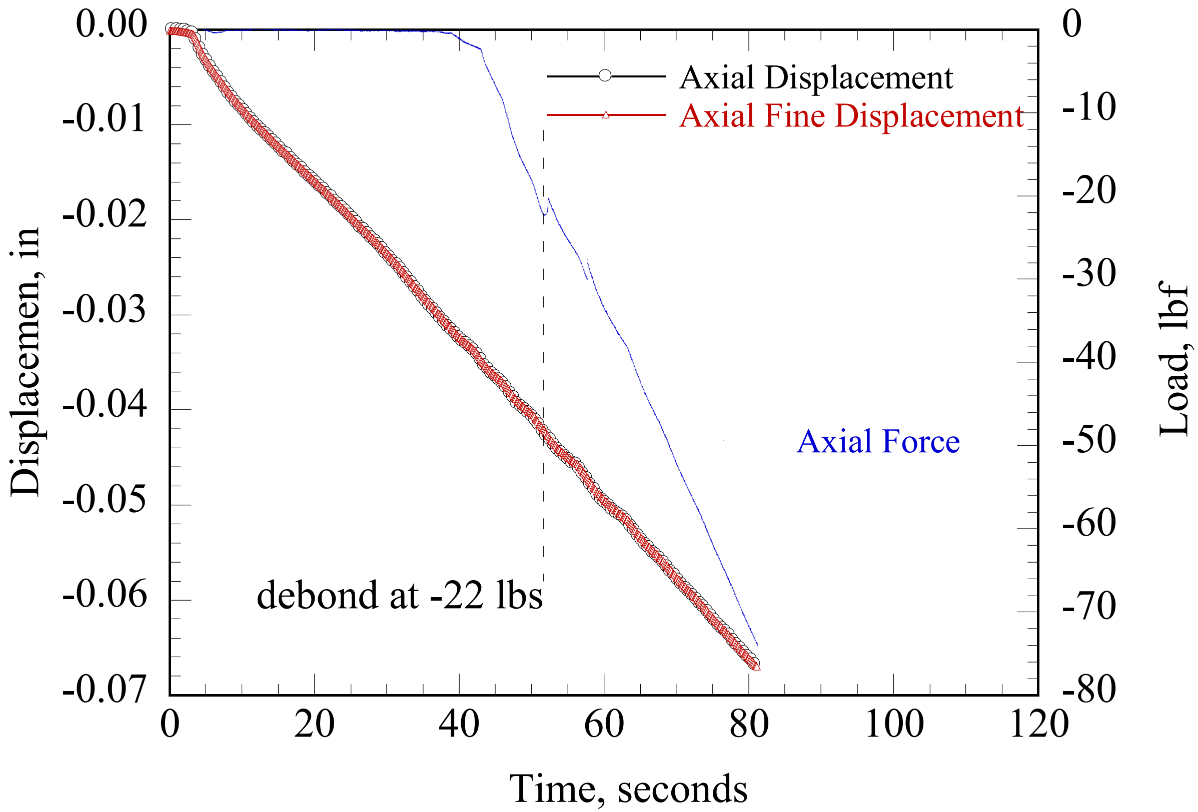


Debond

After test

EBC Interlaminar Strength Test

- 114-93-7-#11 specimen, failure load at -22 and -30 lbf



At -22 lbf (97.882 N)

$$K_I = \frac{F_I(a)P\,(S_o-S_i)}{B\,W^{3/2}} = \frac{1.07*97.882*40*10^{-3}}{(10*10^{-3})*(3.88*10^{-3})^{3/2}*10^{-6}}$$

$$K_I = 1.733\ MPa*m^{0.5}$$

$$K_{II} = \frac{F_{II}(a)P\,(S_o-S_i)}{B\,W^{3/2}} = \frac{0.59*97.882*40*10^{-3}}{(10*10^{-3})*(3.88*10^{-3})^{3/2}*10^{-6}}$$

$$K_I = 0.956\ MPa*m^{0.5}$$

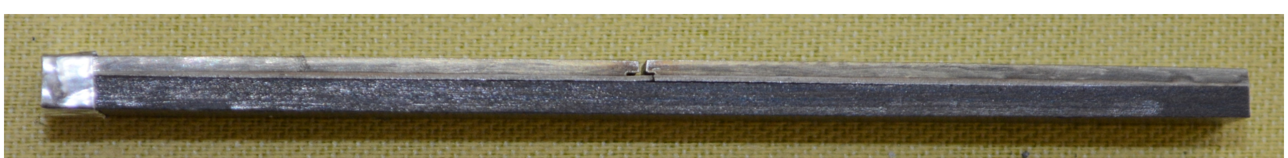
At -30 lbf (133.476 N)

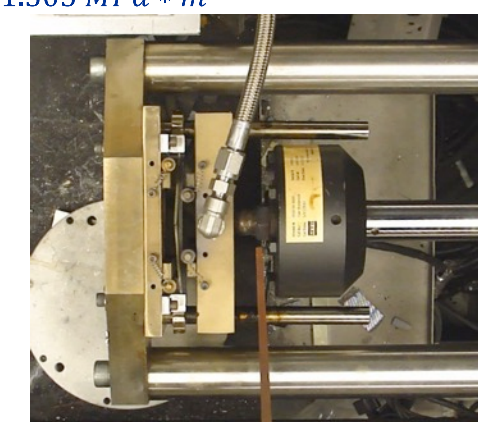
$$K_I = \frac{F_I(a)P\,(S_o-S_i)}{B\,W^{3/2}} = \frac{1.07*133.476*40*10^{-3}}{(10*10^{-3})*(3.88*10^{-3})^{3/2}*10^{-6}}$$

$$K_I = 2.364\ MPa*m^{0.5}$$

$$K_{II} = \frac{F_{II}(a)P\,(S_o-S_i)}{B\,W^{3/2}} = \frac{0.59*133.476*40*10^{-3}}{(10*10^{-3})*(3.88*10^{-3})^{3/2}*10^{-6}}$$

$$K_I = 1.303\ MPa*m^{0.5}$$





Debond